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THE MEASUREMENT OF THERMAL
CONDUCTIVITY AT HIGH TEMPERATURES

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THE MEASUREMENT OF THERMAL CONDUCTIVITY AT
HIGH TEMPERATURES

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SUMMARY

This investigation was conducted to design and construct a device which would not require cumbersome and expensive equipment to measure the thermal conductivity of refractory-type materials at high temperatures and to measure the thermal conductivity of samples of slip cast fused silica.

The guard-ring heater recommended by the American Society of Testing Materials for thermal conductivity tests of refractory-type materials was modified so as to extend its useful temperature range to 2000° F. This was accomplished by modifying the design of the heater plate and power supply, using Kanthal A-1 resistance wire, and insulating the thermocouples in the surface of the plate.

The performance of the equipment was tested by measuring the thermal conductivity of a material of known thermal conductivity. The results of this test indicate that the equipment will produce satisfactory results provided that good surface contact is made between the heaters and samples.

The method employed to measure power in this device is unique for this type of equipment. A calibrated resistance shunt constructed of constantan wire was placed in series with the main circuit heater windings and the voltage drop across this shunt was measured with a potentiometer. This gave current measurements which were very accurate. The voltage drop across the test area of the main heater was measured with

a potentiometer by using a Leeds and Northrup volt-box which reduced the input voltage by a known constant with an accuracy of ± 0.02 per cent.

The surfaces of the slip cast fused silica samples could not be made with the tolerances specified in the A.S.T.M. Standards publication. The error that this introduces in the measurement of the thermal conductivity was not determined, but it can be assumed that the end result was to lower the measured values of thermal conductivity. It is recommended that several samples of slip cast fused silica which have been ground and lapped to A.S.T.M. specifications be tested in this apparatus in order to evaluate the error resulting from poor surface contact.

CHAPTER I

INTRODUCTION

The work being conducted by the Chemical Sciences Division of the Georgia Institute of Technology Engineering Experiment Station with compounds of fused silica required that the thermal conductivity of these various compounds at mean temperatures up to 2000° F. be known.

Many types of devices for measuring thermal conductivity have been constructed and used to obtain data up to a mean temperature of 1600° F. Only a few devices have been built to obtain thermal conductivities of materials above this mean temperature.

At lower temperatures the most widely used device is a guard-ring heater. It was probably first applied by Berget (1)* in 1887 in experiments on mercury and later adopted by Poensgen (1) in 1912 for thermal conductivity measurements of non-metallic substances. In the design of this device a separate heating ring at the outer portion of a heater plate was utilized to reduce the edge losses from the main heating section.

The American Society of Testing Materials (2) has recommended the use of the guard-ring type heater in the testing of refractory materials. The author undertook to modify the design of this heater and its power supply so as to extend its useful temperature range to 2000° F. and to incorporate it into a single piece of permanent equipment for measuring

*Numbers in parentheses following names of individuals refer to the bibliography.

thermal conductivity of refractory types of materials.

The need for a simple device which would not require cumbersome and expensive equipment to measure the thermal conductivity of various refractory-type materials prompted this work.

CHAPTER II

INSTRUMENTATION AND EQUIPMENT

General.--The complete unit is shown in Fig. 1. The rubber hoses are water supply lines to the cooling plates. A constant pressure head was maintained by a water reservoir located near the ceiling of the room. The covered tub on the rear of the control panel houses the heater plates, samples, and insulation. The volt box and potentiometer appear on the shelf below the control panel. The construction details of the cooling plates are shown in Fig. 4.

Direct current circuits.--The guard and main circuits of the main heater plate were supplied with DC power by a compound-wound generator driven by a three phase induction motor. The complete diagrams of these two circuits appear in Figs. 5 and 6.

The guard circuit incorporated three rheostats in series with the resistance windings for control. Two of these rheostats were slide wire resistances of 65 ohms each for rough adjustments. The third rheostat was a 12.5 ohms, circular type for fine adjustments. The maximum current in the guard ring was limited to 3.3 amperes by the ratings of the control rheostats. However, the edge losses from the heater plate were small and the maximum current required at elevated temperatures was approximately 2.0 amperes.

The main circuit utilized three slide-wire rheostats in parallel for rough adjustments and a 12.5 ohm circular rheostat in series with

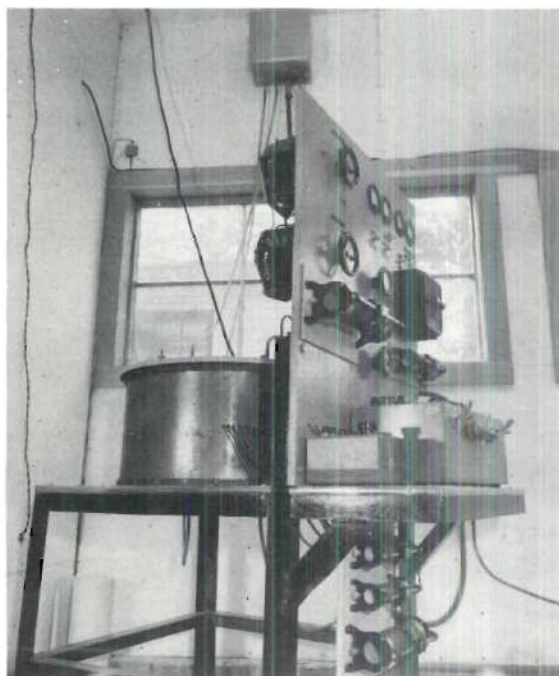


Figure 1. View of Thermal Conductivity Apparatus.

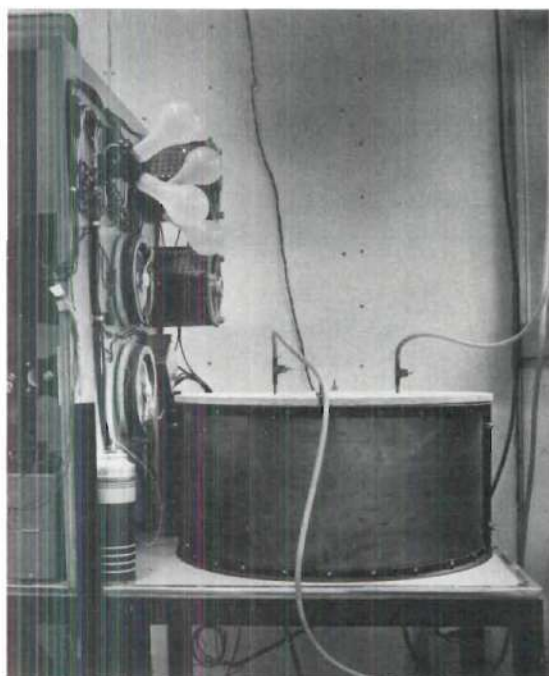


Figure 2. Side View of Apparatus Showing Removable Half of Tub.

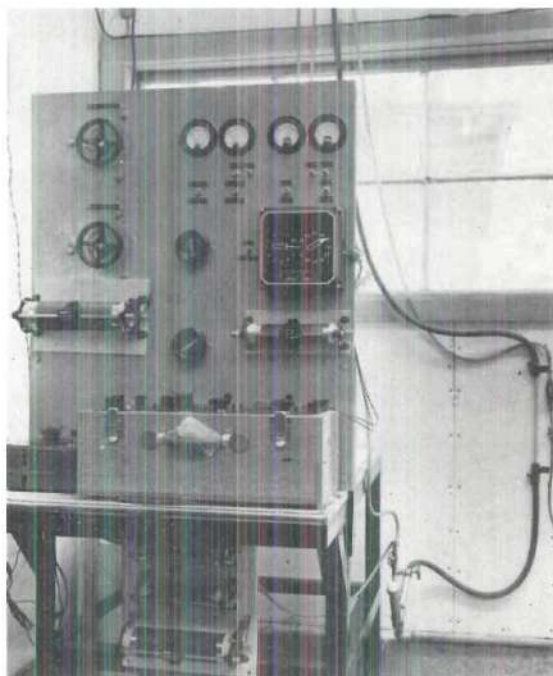


Figure 3. Front View of Apparatus.

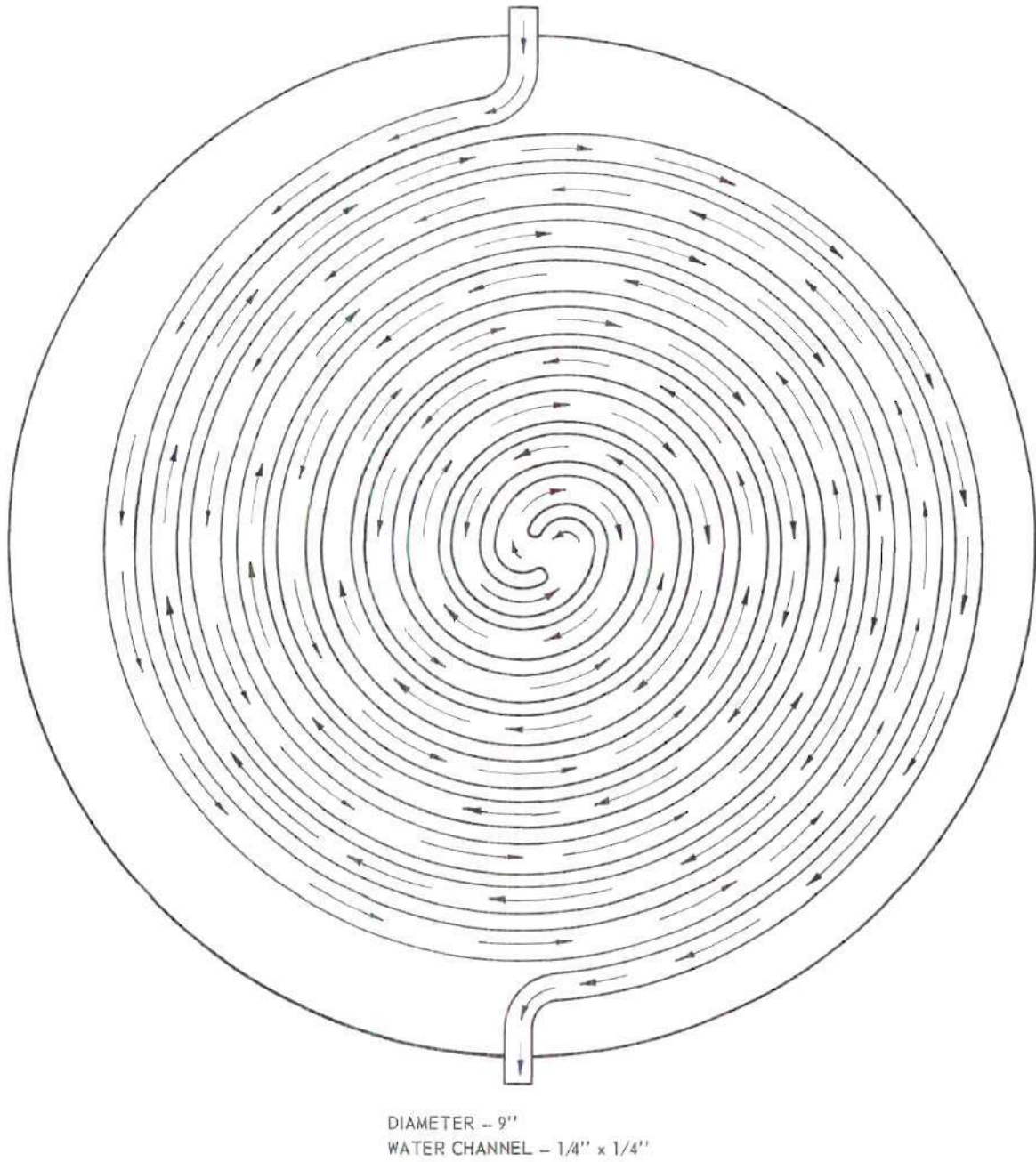


Figure 4. Top View of Cooling Plate with Cover and Gasket Removed.

the load for fine adjustments. The resistance of the three rheostats for rough adjustments was 650 ohms each with a maximum current rating of one-half ampere each. This limited the maximum current in the main circuit to 1.5 amperes. At the upper temperatures the current required in this circuit was approximately 1.4 amperes with a temperature drop of fifty-one degrees across a sample thickness of one half inch.

A calibrated resistance shunt in series with the main circuit heater windings served as a convenient means of accurately measuring the current. The shunt was constructed of constantan wire which has a resistance-temperature coefficient of $0.000002/(^{\circ}\text{C})$ at 25°C . The diagram of this shunt and its voltage measurement leads going to a selector switch are shown in Fig. 6.

The voltage drop across a known area of the main heater was measured by voltage leads imbedded in the surface of the heater plate. These leads were connected through a Leeds & Northrup volt-box which reduced the voltage by a known constant. The voltage output of the volt-box was measured by a potentiometer with an accuracy of ± 0.02 per cent. This voltage across the test area ranged from five volts at relatively low heater plate temperatures to thirty-five volts at the higher temperatures of the heater plate. Thus, the power, $E I$, passing through a known area was easily determined by two settings of a selector switch, measuring at one position the current, and at the other, the voltage drop across the test area.

Considerable difficulty in achieving steady state conditions was experienced during initial operation of the equipment. This was caused by a variation in the generator output voltage. Two modifications

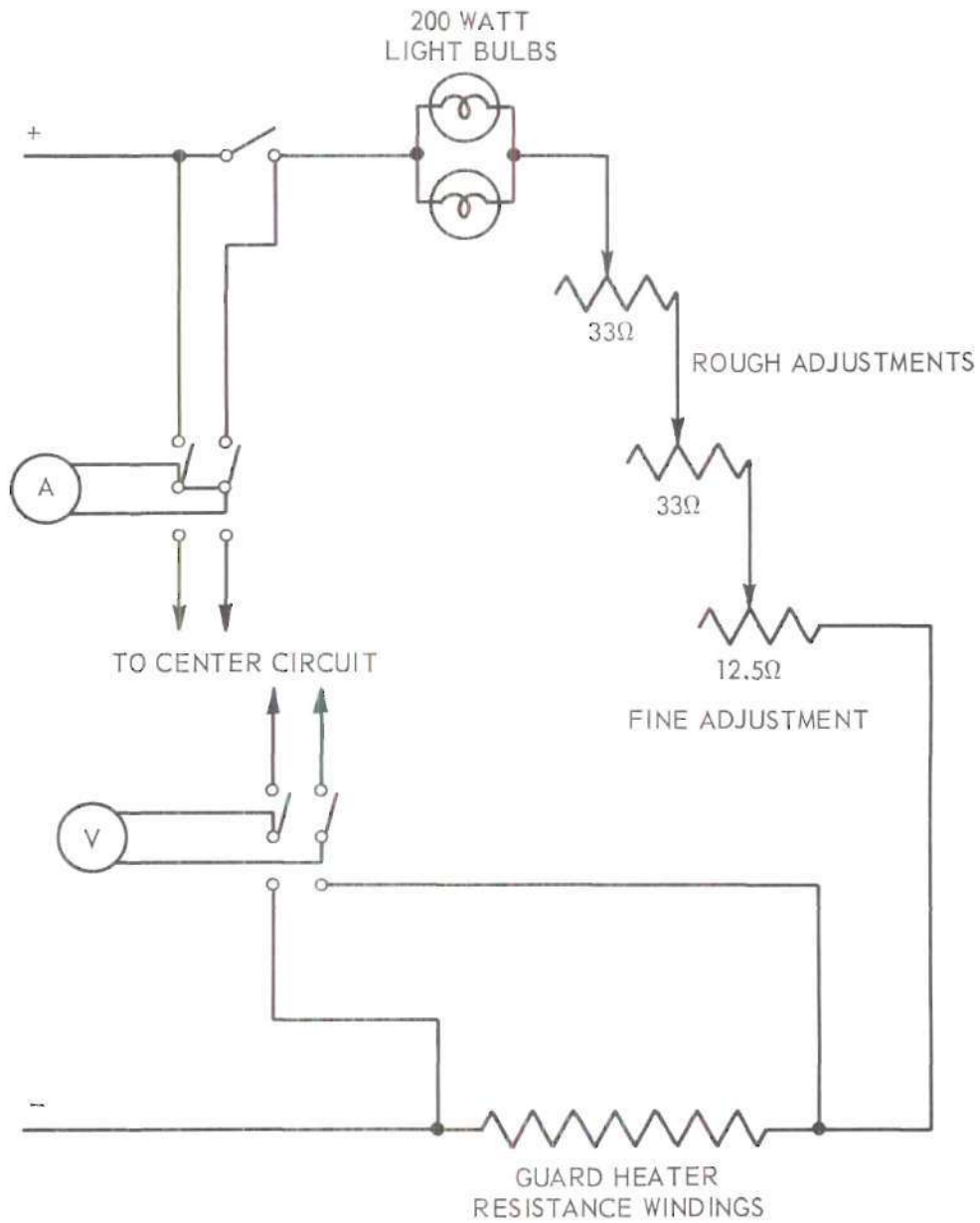


Figure 5. Center Heater Plate Guard Circuit.

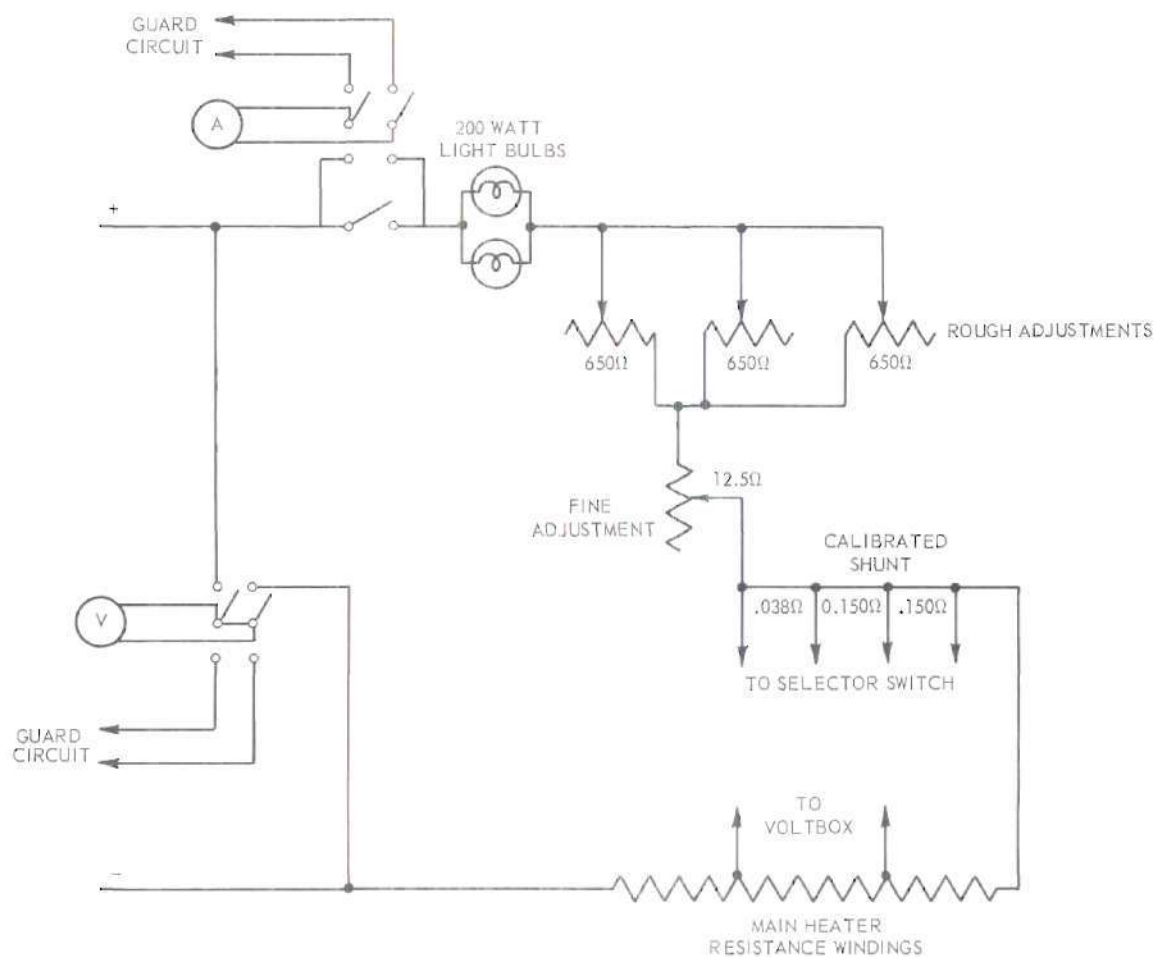


Figure 6. Center Heater Plate Center Circuit.

of the equipment eliminated this problem. These are (a) decreasing the resistance in the generator field so that it was operating at its most stable point, (b) placing two two-hundred watt light bulbs in parallel in each of the two DC circuits. The filaments of these bulbs appeared cherry red since only a small current was flowing in each one. Under these conditions, the filament was very sensitive to any temperature change and acted as a variable resistance in the circuit.

Alternating current circuits.--The two auxiliary heaters were supplied by one hundred and fifteen volts AC power. Each of these plates utilized a powerstat for voltage control to the resistance windings. The two powerstats were connected in parallel to an automatic voltage regulator which maintained a constant one hundred and fifteen volt output.

Isothermal surfaces.--In order to insure that the heat flow through the test sample over the test area was normal to the surfaces of the sample, nickel plates 0.250 inches thick and nine inches in diameter were placed between each of the auxiliary heaters and the low temperature surfaces of the test samples. A soft asbestos sheet, one-thirty-secondth of an inch thick, was placed between the nickel plate and the test sample. This allowed a thermocouple of small wire to be placed on the test sample without completely disrupting the surface contact. The nickel plates, having a high thermal conductivity, insured that at least the test area of the sample was at a constant temperature. The positions of the main heater plate, samples, nickel plates, and auxiliary heaters are shown in Fig. 7.

Temperature Measurement.--Six cromel-alumel thermocouples, B. and S. 24 gage, were used to measure temperatures. Four of the thermocouples were imbedded on one side of the main heater plate along a radius of the plate. These four thermocouples were placed so that two were over the guard circuit and two were over the center circuit. The temperatures of the two low temperature surfaces of the samples were measured by placing a thermocouple on the center of each sample and insulating it from the nickel plate by a soft asbestos sheet. The recommended maximum service temperature for this size and type of thermocouple was exceeded during operation of the apparatus (3). This required replacement of all thermocouples after the three tests reported in this investigation were completed.

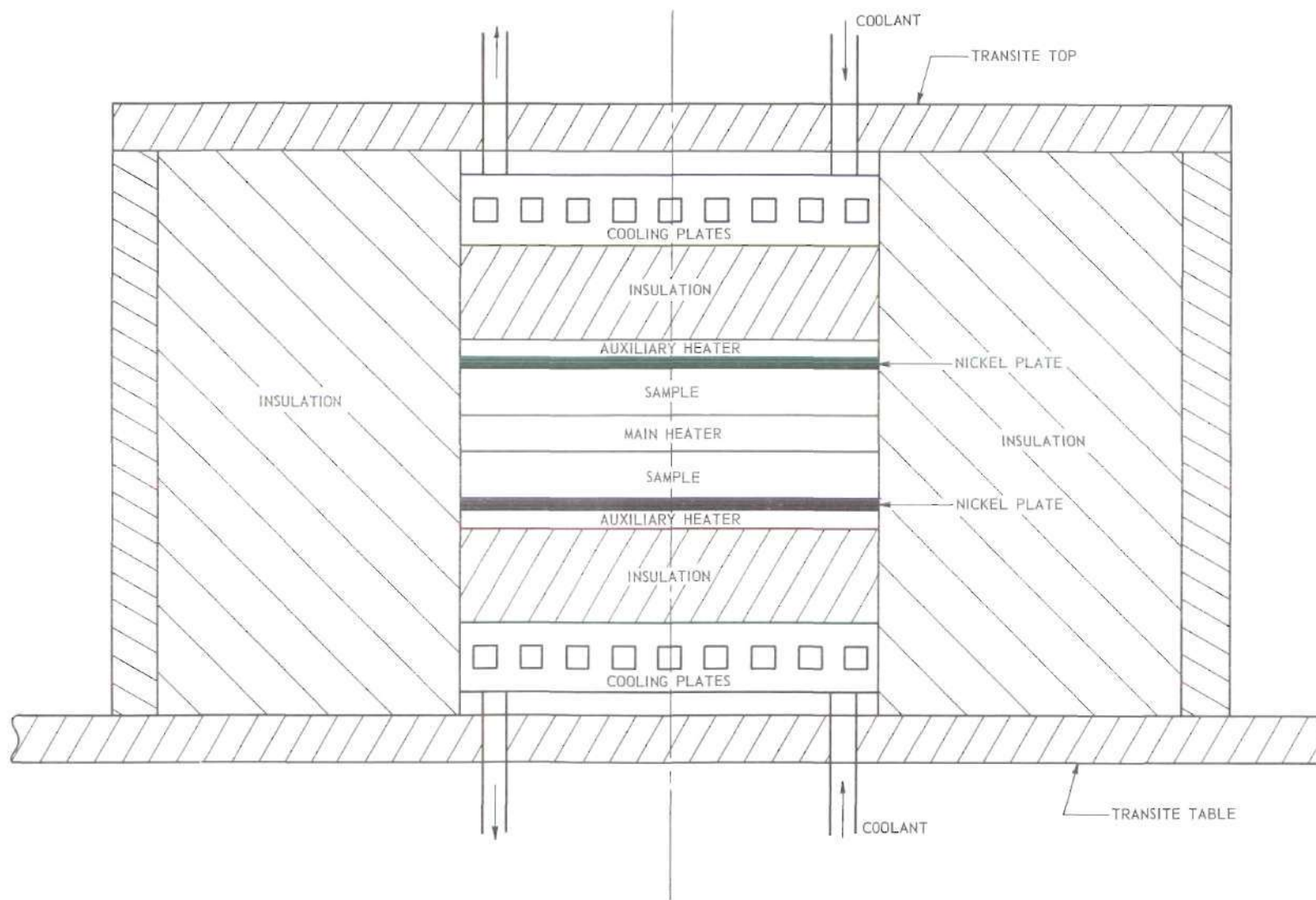


Figure 7. Sectional View of Tub.

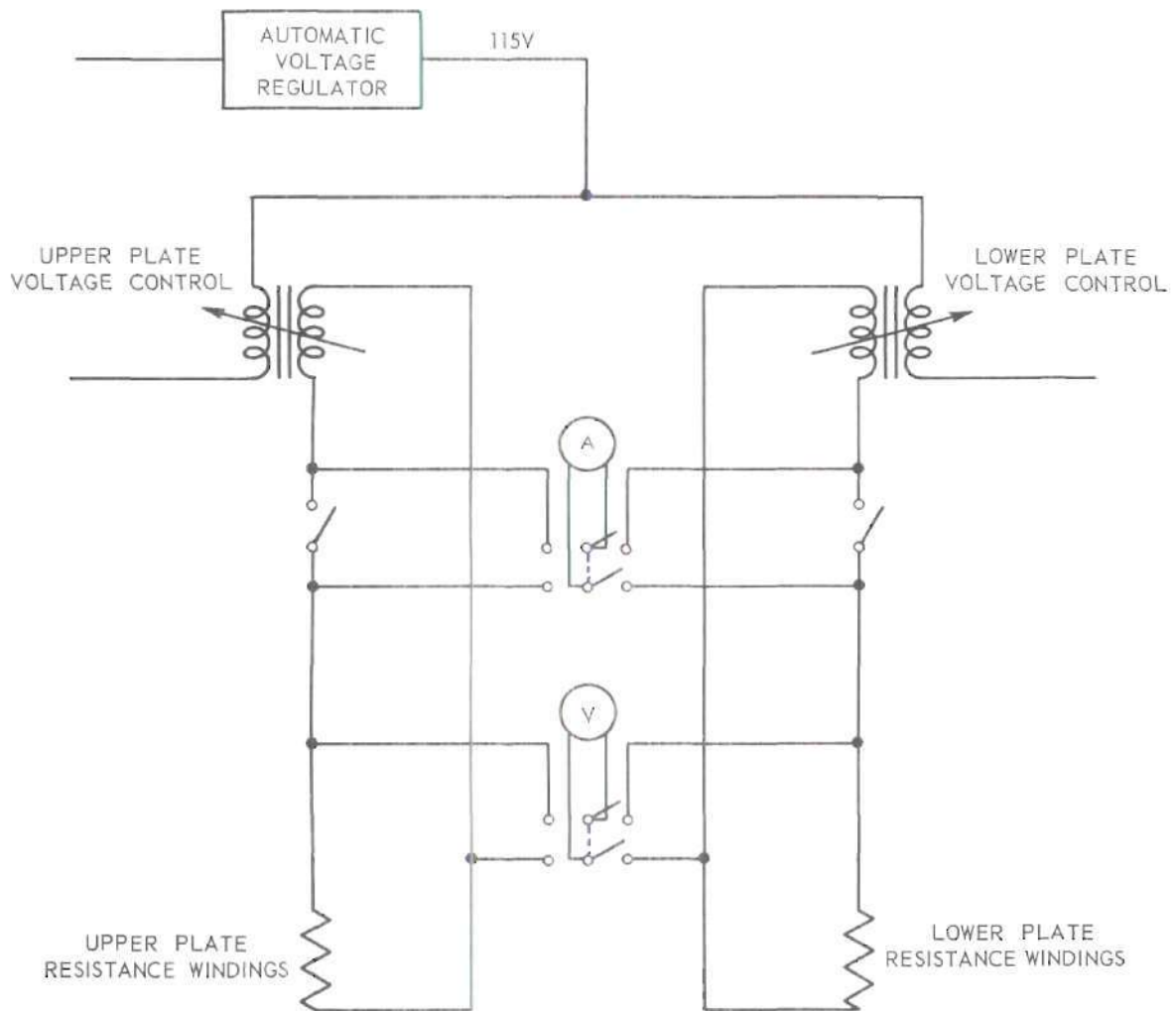


Figure 8. Auxiliary Heater Plate Circuits.

CHAPTER III

HEATER PLATE CONSTRUCTION

Main heater plate.---The heater plates used are of the "Alundum" type which were originally developed by the Mellon Institute of Industrial Research. The percentage composition by weight of the plastic clay used is as follows:

Kaolin (Putnam)	30
Potter's Flint	20
Feldspar (Woodcox)	35
Ball Clay (Kentucky #12)	<u>15</u>
	100

Twenty per cent by weight of the above mixture was used with a mixture of 70 per cent of 38-60 mesh Alundum and 30 per cent of 180 mesh Alundum. These compositions were combined with 22 per cent by weight of water to form a thick paste and cast in a wax mold.

The technique of construction of the wax mold is given in detail since the evolution of the technique required substantial trial and error testing.

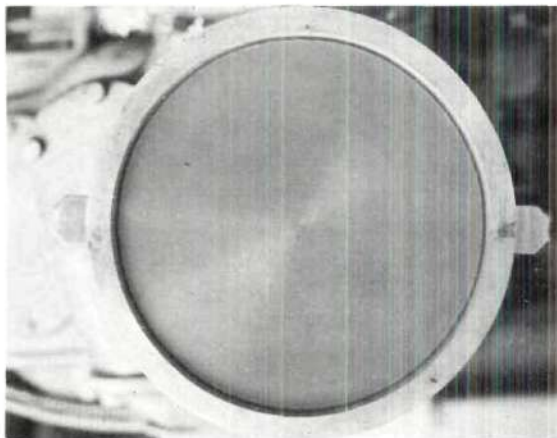
An aluminum disc ten inches in diameter and one inch thick was clamped in a lathe and recessed to a depth of 0.438 inches and 9.250 inches in diameter. The aluminum plate was removed from the lathe and the recess was filled with a mixture of one part Gulf Petrowax A and one part paraffin wax as recommended by the A.S.T.M. Standards publication.

After the wax had solidified the aluminum plate was centered in the chuck of the lathe and a depression cut into the wax to a depth of 0.250 inches and a diameter of 9.125 inches. The depression was increased to a total depth of 0.350 inches at the outer circumference of the 9.125 inch diameter recess by cutting a groove 0.094 inches wide and 0.100 inches deep at the outer circumference of the recess. A double spiral groove of ten turns per inch with a width of approximately 0.060 inches was then cut in the wax to a depth of 0.100 inches.

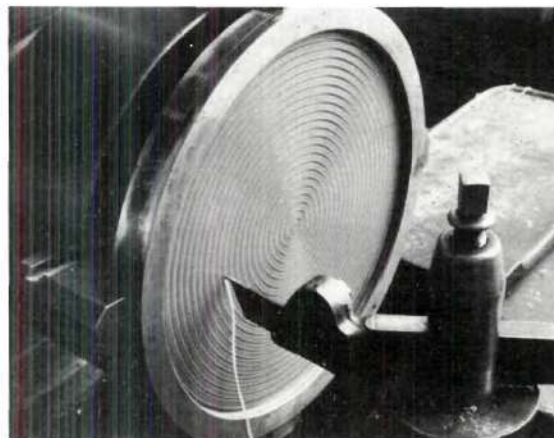
In cutting the spiral grooves the tool shown in Fig. 9 was used. Four ribbons of wax 0.025 inches thick were cut from each groove from the outside of the plate toward the center as shown in Fig. 9(B). After cutting one groove to the prescribed depth the tool was repositioned 0.100 inches toward the center and the second spiral cut between the grooves of the first. On each cut the small end of the wax ribbon which stuck to the center of the plate was removed by hand using a small piece of wire sharpened on one end. This prevented a build-up of wax in the grooves at the center.

The wax mold was then placed on a mechanical vibrator and the mixture for the aluminum plate was poured very slowly into the mold. The vibration allowed the grooves in the wax to fill without trapping air bubbles. The mold was filled completely, covered with a damp cloth and dried at 125° F. for eighteen hours. It was then placed in a high temperature dryer at approximately 220° F. for eight hours.

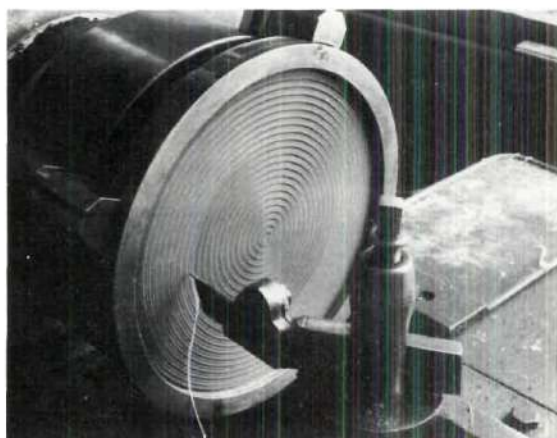
After this drying period the aluminum plate was carefully removed from the aluminum plate and placed grooved side up on a flat surface. It was then placed in a furnace and fired to 2000° F.



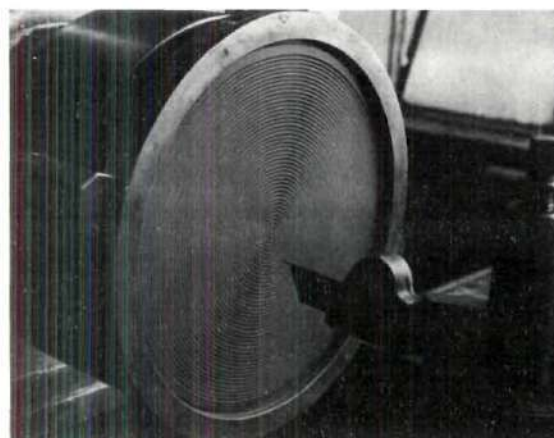
A. Wax filled mold with circular groove.



B. Cutting first spiral.



C. Cutting second spiral between first spiral grooves.



D. Completed wax mold.

Figure 9. Steps in Producing Wax Mold.

The alundum plate was then grooved for thermocouples and voltage and power leads. The back surface of the plate was then ground with an aluminum oxide surface to produce a surface as flat as possible. After all cutting operations were completed, the plate was again fired to 2540° F.

A cover plate for the main heater was made in a similar manner. A single recess 0.250 inches deep and 9.125 inches in diameter was cut into a wax filled aluminum plate and filled with the alundum mixture. This cover plate was cemented to the main heater by Sauereisen High Temperature INSA-LUTE Cement after the heater plate was completely wired with Kanthal A-1 wire.

Auxiliary heaters.--Two auxiliary heaters were made following the procedure already outlined with one exception. There was no need to produce two identical surfaces on these heaters as was the case with the main heater; therefore, the cover plate was not used. Instead the resistance windings were covered with Sauereisen High Temperature INSA-LUTE Cement and were ground flat after drying.

CHAPTER IV

PROCEDURE

Operation of equipment.--The possibility of irreversible chemical changes occurring in the test sample required that the determination of the thermal conductivity be done in steps of increasing temperature. The requirements that steady state be achieved and that the temperatures be adjusted within very close limits at each step represented the most difficult part of the operation.

In starting a test run all the heaters were placed in operation at a low power setting and the valves in the water lines through the cooling plates were opened an equal amount. During the first several hours of this initial warm-up period the temperature gradient across the guard circuit area and the main circuit area of the main heater was checked frequently to eliminate the possibility of cracking the heater plate.

Once the temperature change with time became small, minor adjustments were made in the power settings of the various heaters every few hours. These adjustments were made in order to balance the temperatures across the guard and main circuit areas of the main heater and to balance the temperatures of the cold surfaces of the two samples. When these temperatures were equal and there was no change in temperature with time, the measured values of the current, the voltage drop, and the surface temperatures were recorded.

Equalizing the temperatures of the cold surfaces of the two samples was the most difficult single adjustment. For this reason if steady state was reached with a temperature difference of one or two degrees between the two cold surfaces of the samples and the temperatures between the guard and main circuit areas were equal, the measured heat flow was divided in proportion to the temperature drop across the samples.

Performance.--A sample of known thermal conductivity was used to evaluate the overall performance of the equipment. This sample was a silica base solid insulation with a trade name of "Min-k 1301". The k values appearing in Fig. 10 for this known sample were guaranteed by the manufacturer to be within ten per cent of the actual thermal conductivity.

The results obtained with the instrument described above with the sample of known thermal conductivity are shown in Fig. 10 along with the values furnished by the manufacturer. The measured values are within the limits of accuracy given by the manufacturer. However, the performance of the equipment was largely dependent upon the surface finish of the samples. Excellent surface contact was made using the "min-k 1301" samples. On the other hand, the surface finish of the slip cast fused silica sample could not be produced with the tolerances specified by the A.S.T.M. Standards publication (2).

Routine calculations.--The equation used to calculate the thermal conductivity is

$$k_m = C \frac{E I}{2 A} \frac{T}{(t_1 - t_2)}$$

where C = dimensional coefficient

E = voltage across test area

I = current in the main circuit

T = thickness of the test samples

A = area of the test section

t_1 = temperature of the test area

t_2 = temperature of the cold surface of the test sample

k_m = thermal conductivity at the arithmetic mean temperature,

$$\frac{t_1 + t_2}{2}$$

In the particular case when the temperature of the cold surfaces of the samples differed by one or two degrees the following equation was used:

$$k_m = C \frac{E I}{1 + \frac{t_1 - t_2}{t_1 - t_3}} \frac{T}{A (t_1 - t_3)}$$

where t_2 and t_3 are the temperatures of the cold surfaces (4).

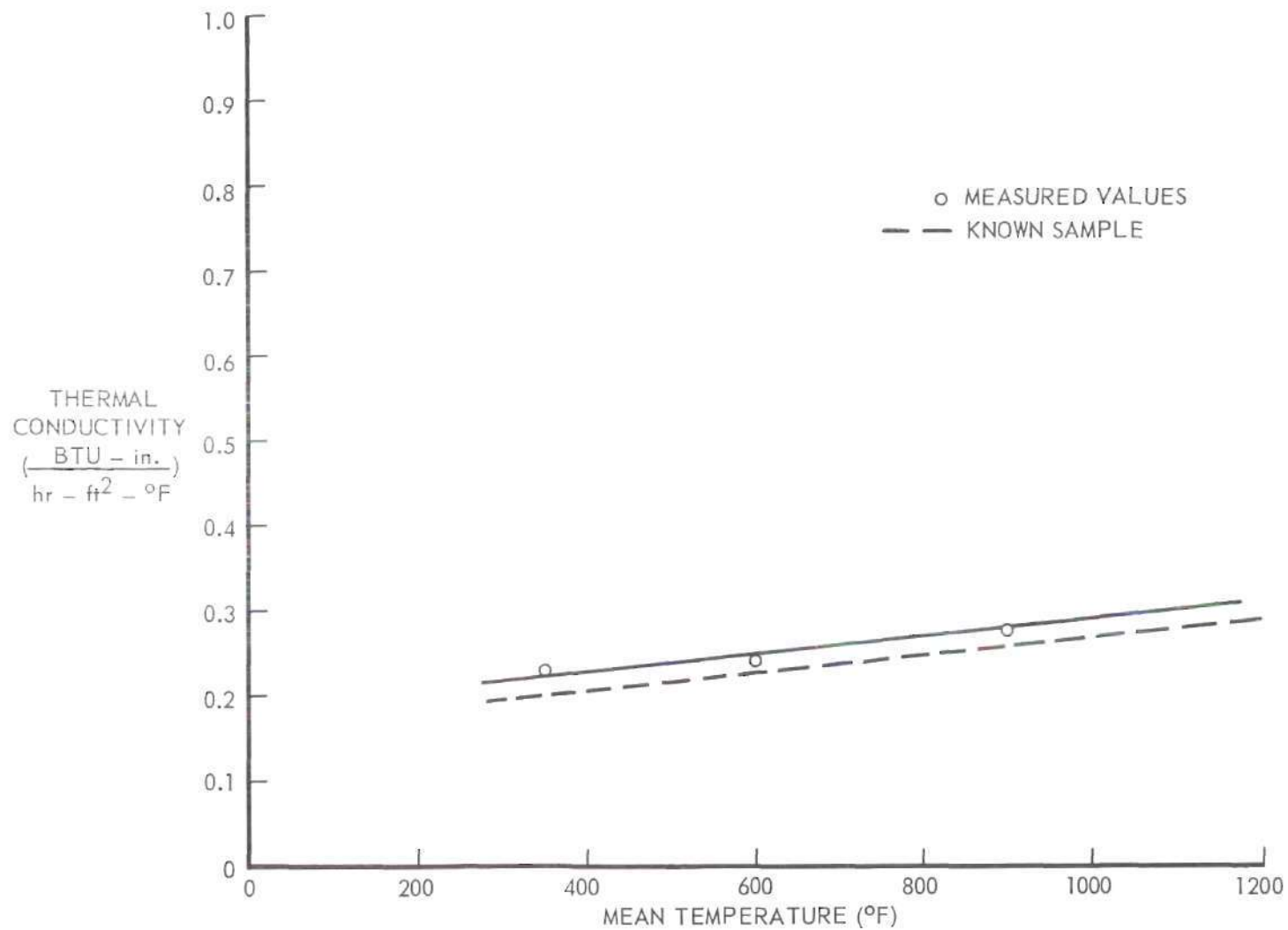


Figure 10. Comparison of a Sample of Known Thermal Conductivity with Measured Values.

CHAPTER V

DISCUSSION OF RESULTS

The k values measured using the one-quarter inch fused silica samples differ by approximately 7 per cent from the values determined with the one-half inch samples as shown in Figs. 11 and 12*. The difference is attributed to the smaller temperature drop across the thinner sample which resulted in a larger possible error in temperature drop measurements, and also to the small differences in particle size, per cent solids, etc., in the two different slips used to make the samples. A complete description of the two slips used is given in the appendix.

The results of the performance test using "Min-k 1301" indicate that the apparatus will produce satisfactory results provided that good surface contact is made between the heaters and samples. The surfaces of the slip cast fused silica samples could not be made with the tolerances specified in the A.S.T.M. Standards publication. The error that this introduced was not determined, but it can be assumed that the end result was to lower the measured values of thermal conductivity.

The determination of a "maximum" error is given in Appendix A. This was calculated to be 5.6 per cent. This result is based only upon the possible errors in determining each of the terms used in the equation for calculating the thermal conductivity. Omitted from this

* Later work has indicated that firing time and temperature will significantly affect the thermal conductivity of slip cast fused silica. An investigation of this effect is being conducted at the present time.

result are the possibility of edge losses in the samples, incomplete contact with all the surfaces of the samples, small changes in room temperature, and small changes in the temperature of the incoming cooling water. These effects were assumed to be negligible.

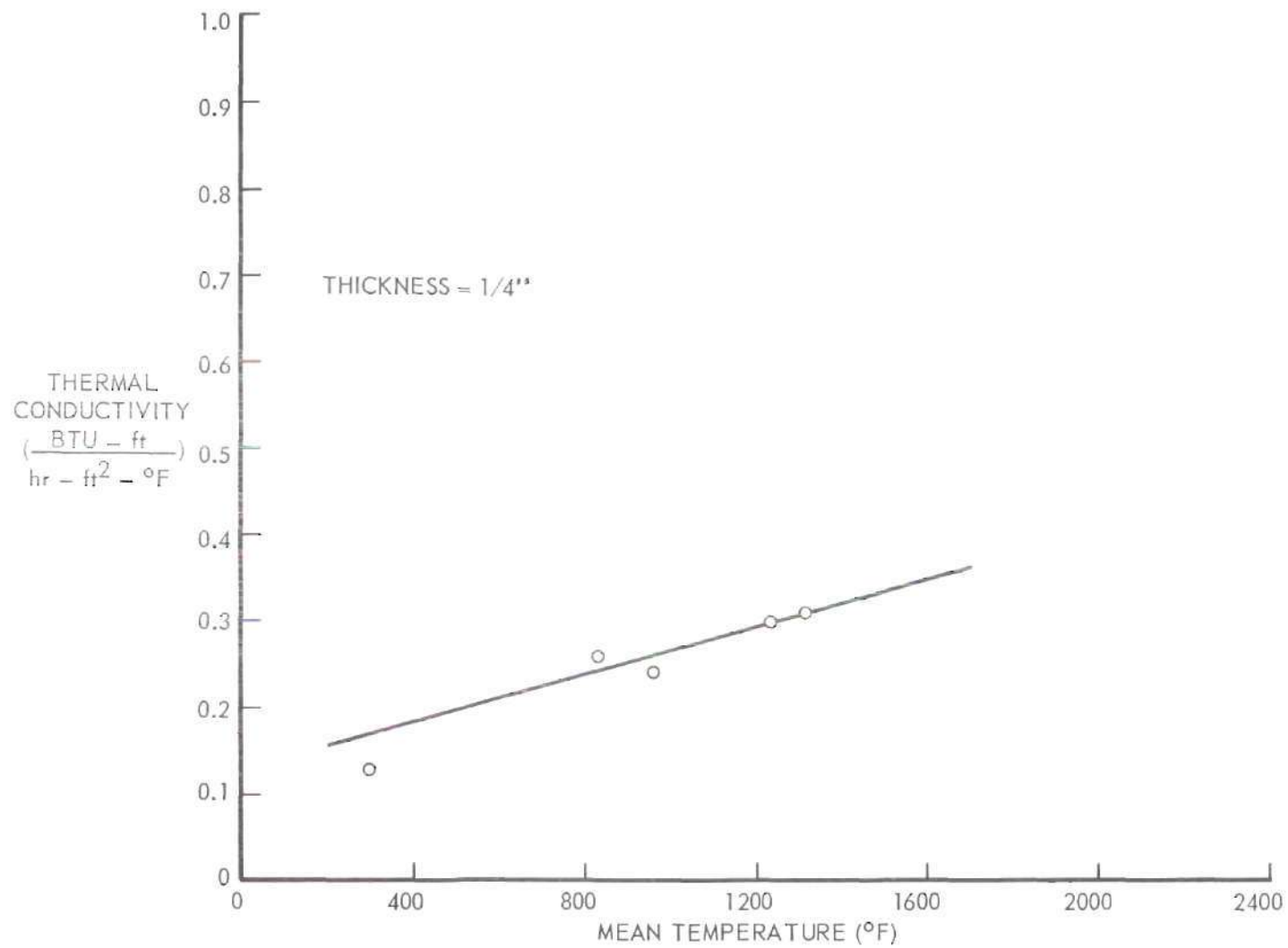


Figure 11. Thermal Conductivity of a Slip Cast Fused Silica Sample.

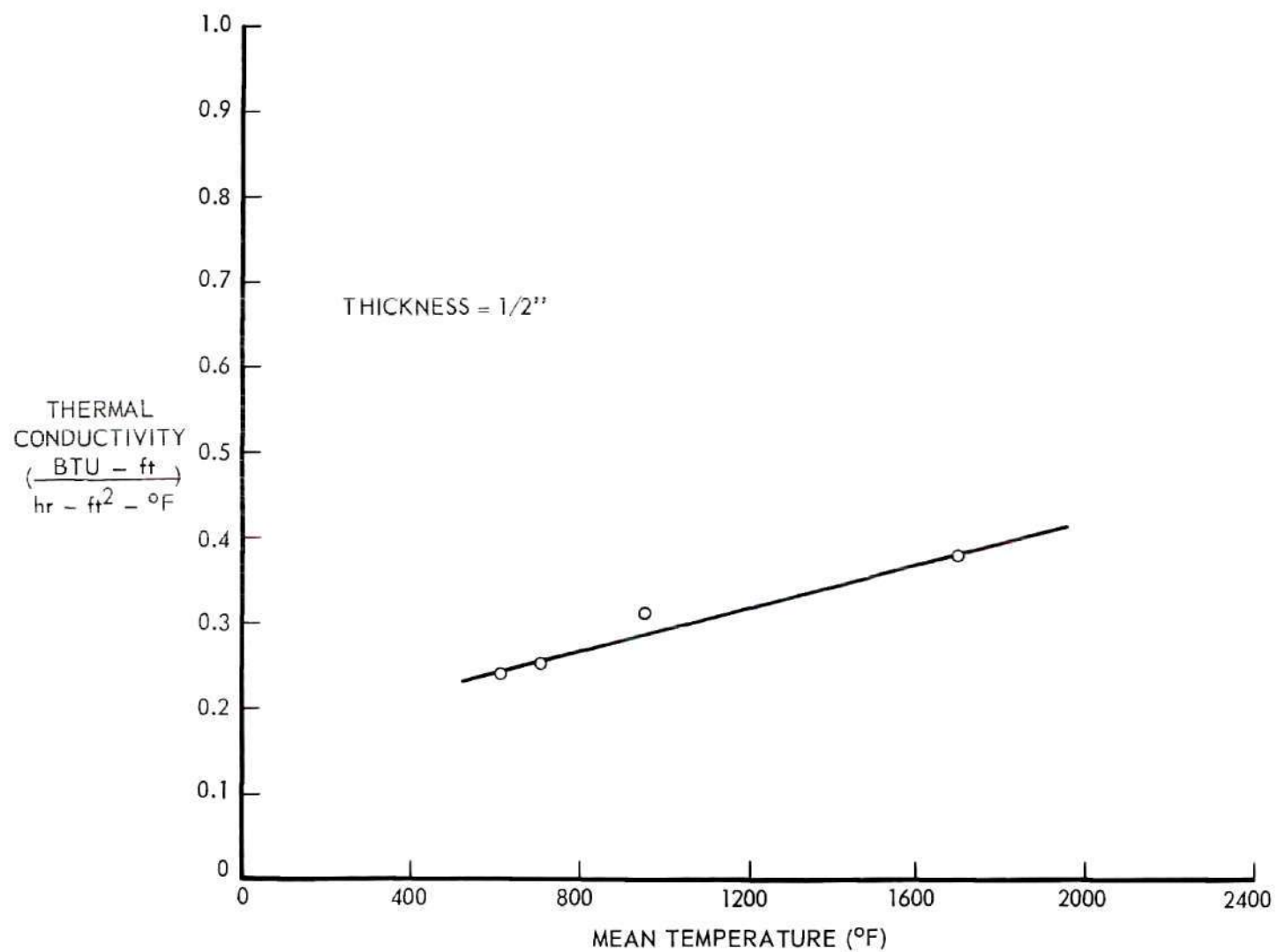


Figure 12. Thermal Conductivity of a Slip Cast Fused Silica Sample.

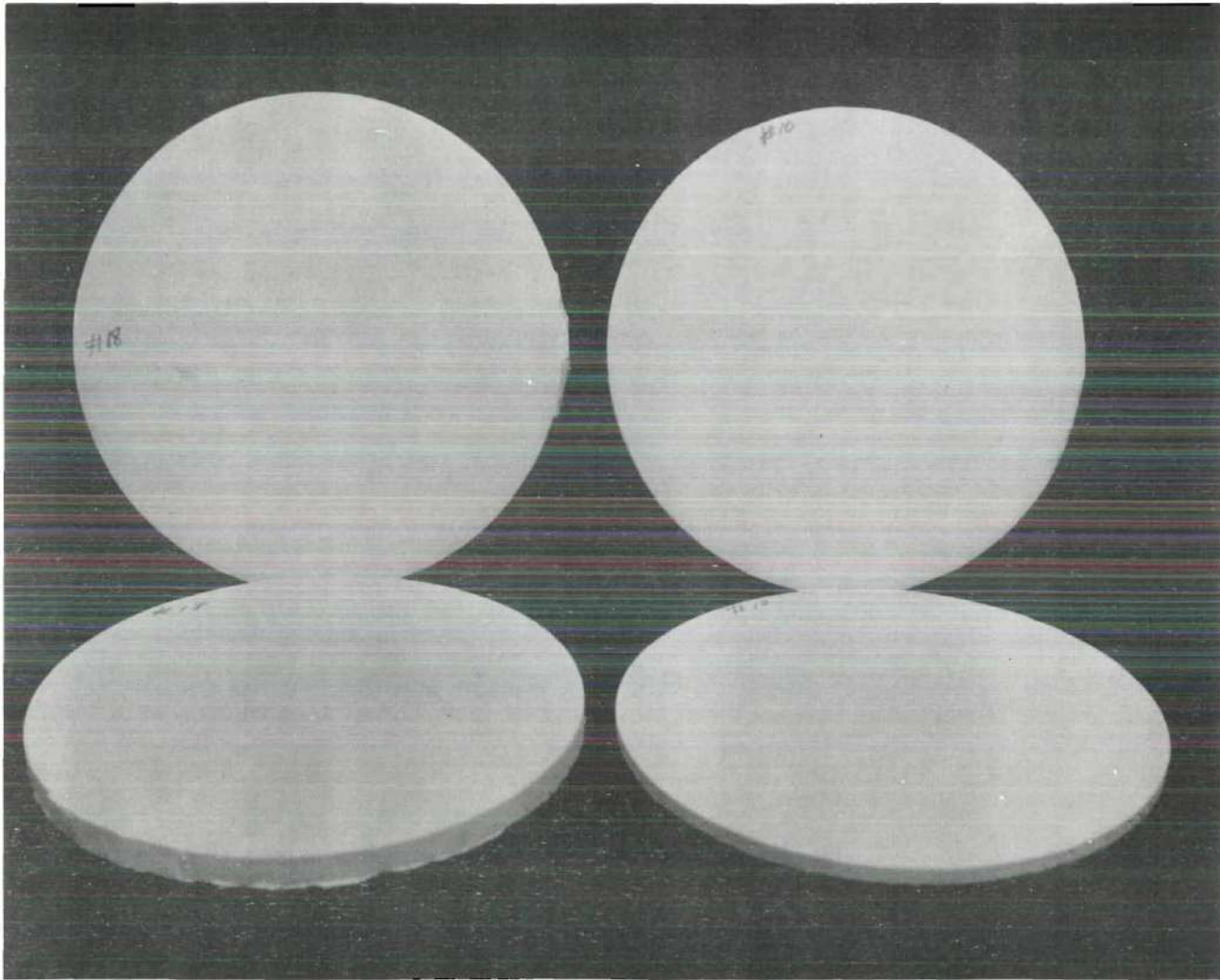


Figure 13. Samples of Slip Cast Fused Silica.

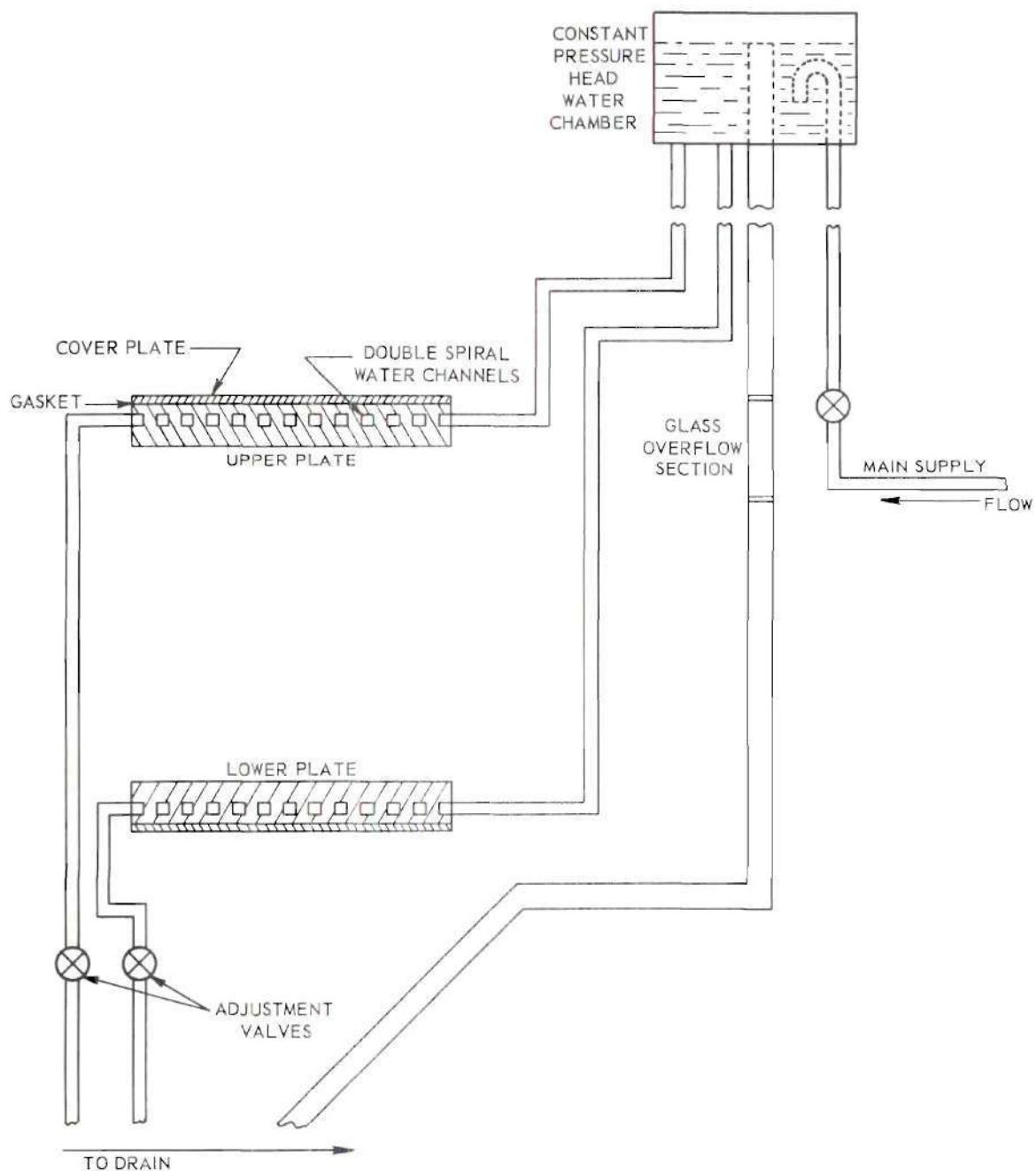


Figure 14. Schematic Diagram of Cooling System.

CHAPTER VI

CONCLUSIONS AND RECOMMENDATIONS

The performance of the apparatus at temperatures above 1600° F. indicate that no leakage to the thermocouples occurred. This is attributed to the low power requirements of the main heater, the increased thickness of the heater plate, and the insulation of the thermocouples in the surface of the main heater.

The method used to measure power is unique in this type of equipment. The use of a calibrated constantan wire shunt with a relatively inexpensive Leeds and Northrup volt-box gave power measurements which were extremely accurate. The economy and accuracy of this method of power measurement made it superior to the usual technique of using wattmeters.

The results obtained with the one-quarter inch sample of fused silica are not considered satisfactory since the temperature drop across the sample was small. The values measured with the one-half inch samples should more accurately represent the temperature-thermal conductivity relationship for slip cast fused silica. However, the measured thermal conductivity of this material is probably lower than the actual value due to the unsatisfactory surface finish of the samples. It is recommended that several samples of slip cast fused silica be ground and lapped to A.S.T.M. specifications and tested in this apparatus in order to evaluate the error resulting from poor surface contact.

The operation of the apparatus was time consuming and difficult. This was primarily due to difficulties in balancing cold surface temperatures and achieving steady state. This operation would be greatly enhanced by installing in the auxiliary heater plate circuits a more sensitive control than the powerstats.

APPENDIX A

MAXIMUM ERROR

The equation for calculating the thermal conductivity is

$$k_m = C \frac{E}{2A} \frac{I}{T} \frac{T}{\Delta t}.$$

From this equation the total derivative is

$$dk_m = \frac{\partial k_m}{\partial E} dE + \frac{\partial k_m}{\partial I} dI + \frac{\partial k_m}{\partial T} dT + \frac{\partial k_m}{\partial A} dA + \frac{\partial k_m}{\partial (\Delta t)} d(\Delta t)$$

$$\begin{aligned} dk_m &= \frac{CIT}{2A \Delta t} dE + \frac{CET}{2A \Delta t} dI + \frac{CEI}{2A \Delta t} dT + (-1) \frac{CEIT}{2A^2 \Delta t} dA \\ &\quad + (-1) \frac{CEIT}{2A (\Delta t)^2} d(\Delta t) \end{aligned}$$

Substituting into this expression and factoring yields:

$$dk_m = k_m \left[\frac{dE}{E} + \frac{dI}{I} + \frac{dT}{T} - \frac{dA}{A} - \frac{d(\Delta t)}{\Delta t} \right]$$

$$\frac{dk_m}{k_m} = \frac{dE}{E} + \frac{dI}{I} + \frac{dT}{T} - \frac{dA}{A} - \frac{d(\Delta t)}{\Delta t}$$

The error computed in this manner is a maximum when all the terms on the right add. Thus the maximum error can be expressed in the following way:

$$\text{maximum error} = \pm \left[\frac{dE}{E} + \frac{dI}{I} + \frac{dT}{T} + \frac{dA}{A} + \frac{d(\Delta t)}{\Delta t} \right]$$

In order to determine a numerical value from this expression, a value for the temperature drop, Δt , across the test samples must be assumed. The A.S.T.M. recommended minimum Δt of thirty degrees is used in order that

$$\frac{d(\Delta t)}{(\Delta t)}$$

be a maximum. The error in measuring both E and I is so small it is neglected.

Substituting the numerical value of the possible error in each term into the maximum error expression yields:

$$\text{maximum error} = \pm \frac{.003}{.500} + \frac{1.5}{30} \times 100 \text{ o/c}$$

$$\text{maximum error} = \pm 5.6 \text{ o/c}$$

APPENDIX B

DESCRIPTION OF SLIPS

	<u>Slip 18</u>	<u>Slip 10</u>
<u>Mill Charge</u>		
Fused Silica	1233 lb. -4+20 mesh	1233 lbs. -4+20 mesh
Water	31 gallons	31 gallons
Grinding Time	16 hours	24 hours
<u>Properties</u>		
Slip Density	1.82 gm/cc	1.82 gm/cc
Per cent Solids	82.5%	82.5%
pH	7.5 - 7.0	7.2 - 6.8
Viscosity	120 - 150 cps	120 - 150 cps
Density after firing	1.85 - 1.95 gm/cc	1.85 - 1.95 gm/cc
Water Absorption	5 - 6%	5 - 6%
Porosity	10 - 14%	10 - 14%
<u>Particle Size:</u>		
Greater than 44 micron	4 - 6%	2 - 4%
Less than 2 micron	23 - 26%	31 - 34%

BIBLIOGRAPHY

1. Jacob, Max, Heat Transfer, Vol. I, John Wiley and Sons, Inc., New York (1949).
2. American Society of Testing Materials Standards, 1955, C177-45.
3. Dike, Paul H., Thermoelectric Thermometry, monograph for Leeds and Northrup Company, Philadelphia 44, Pennsylvania.
4. McAdams, William H., Heat Transmission, McGraw-Hill, New York, (1954).